

## PROJECT DATA

**Bio Process Innovation, Inc. - 03GO13006**

### High Speed / Low Effluent Process for Wet and Dry Mill Corn to Ethanol and Co-Products

<p>Recipient: Bio Process Innovation, Inc</p> <p>Recipient Project Director: Dale Clark 765.746.2100 226 North 500 West West Lafayette, IN 47906</p> <p>Recipient Type: For-Profit Organization</p> <p>Subcontractor(s):</p> <p>EERE Program: Biomass</p>	<p>Instrument Number: DE-FG36-03GO13006</p> <p>CPS Number: 5412</p> <p>HQ Program Manager: Lisa Barnett 202.586.2212</p> <p>GO Project Officer: Andy Trenka 303.275.4745</p> <p>GO Contract Specialist: Melissa Wise 303.275.4907</p> <p>B&amp;R Number(s): ED1906020, W10507000 (HA)</p> <p>PES Number(s): 03-10065, 04-10011</p> <p>State Congressional District: IN - 7</p>
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**PROJECT SCOPE:** This project will demonstrate on a lab and large pilot scale, the High Speed/Low Effluent (HS/LE) fermentation process as it would apply to wet mill and dry mill corn to ethanol. In addition, further evaluation and modeling of MVR distillation to accompany this fermentation process will occur.

#### FINANCIAL ASSISTANCE

Approved DOE Budget:	\$200,000	Approved DOE Share:	\$200,000
Obligated DOE Funds:	\$200,000	Cost Share:	\$410,634
Remaining Obligation:	\$0		
Unpaid Balance:	\$162,101	<b>TOTAL PROJECT:</b>	<b>\$610,634</b>

Project Period: 9/27/03-5/15/05

**TECHNICAL PERFORMANCE**  
**DE-FG36-03GO13006**  
**Bio-Process Innovation, Inc.**  
**High Speed/Low Effluent Process for Ethanol**

**PROJECT SYNOPSIS**

The goal of this project is to demonstrate on a lab and large pilot scale, the High Speed/Low Effluent (HS/LE) fermentation process as it would apply to wet mill and dry mill corn to ethanol. This project will develop and scale up processes for both wet mill (BPI's WM-1 process) and dry mill (BPI's DM-2 process) corn to ethanol which to date have been demonstrated only on the lab and small pilot scale on molasses and corn syrup. It will also determine the steady-state effects of a high degree of recycle and model the incorporation of energy-saving MVR distillation in conjunction with the HS/LE fermentation. Energy savings result from reduced evaporation needs along with a greatly reduced energy usage in distillation. In addition, the project will require further evaluating and modeling of MVR distillation to accompany this fermentation process.

**SUMMARY OF TECHNICAL PROGRESS**

Bio-Process Innovation (BPI) has been in negotiations to use Xethanol Corp. as a prospective site host for the HS/LE pilot trials. Design work has continued on the low energy distillation design project, completing both a rigorous stage-to-stage design of an ethanol distillation column and completing an economic evaluation of column costs at various pressure drops and approach temperatures in the reboiler.

Progress has been made on the saccharification of wet mill syrup, and BPI established the ability of commercial gluco-amylase (GA) to easily and completely convert commercial dextrin syrups to glucose rapidly enough to ensure complete conversion and utilization of the dextrans to glucose then ethanol in the short fermentation cycles characteristic of the HS/LE fermentation of six to twelve hours. A brief hot hold at 55°C with GA prior to fermentation or simultaneous saccharification at fermentation temperatures with slightly higher levels of GA gave complete conversion of the dextrans, allowing a complete fermentation.

A series of tests were run using a high level of back-set in lab-scale fermentations. Long-term trials were performed to test the effects of stillage "back-set" at 60% levels and BPI found no noticeable loss in fermentation rates.

**SUMMARY OF PLANNED WORK**

Bio-Process Innovation will complete the site negotiations for the pilot plant and begin preliminary modifications of pilot reactors for wet mill trials. They will also complete work on the design and optimization of the Low Energy distillation system. Detailed engineering sketches of the LE distillation system will be completed during the next period.

**PROJECT ANALYSIS**

The project is on schedule and on budget. No major obstacles are seen at this time. BPI is applying for a patent on the HS/LE process and, based on the success of this project, may apply for a no-cost time extension, to apply the process to dry mill corn.

A presentation entitled "High Speed Low Effluent Fermentation Process for Dry Mill Corn" was developed and given to ICM, a major dry mill ethanol plant design/build company based in Wichita, KS. It was also sent to Xethanol Corp., as they are starting to run ethanol plants in Iowa to develop a dry starch/fiber separation technology. A poster presentation will be given at the Corn Utilization Conference in June 2004.

**ACTION REQUIRED BY DOE HEADQUARTERS**

No action is required from DOE Headquarters at this time.

**STATEMENT OF WORK**  
**DE-FG36-03GO13006**  
**Bio-Process Innovation, Inc.**  
**High Speed/Low Effluent Process for Ethanol**

**PROJECT GOAL**

The objective of this project is to demonstrate on a lab and large pilot scale, the High Speed/Low Effluent (HS/LE) fermentation process as it would apply to mill and dry mill corn to ethanol. In addition, further evaluating and modeling the use of MVR distillation to accompany this fermentation process will be performed.

**Detailed Task Description**

**Task 1. Wet Mill HS/LE**

**1.0 HS/LE with Wet Mill Syrup**

There are several companies currently combining wet mill corn processing with ethanol production. Wet mill processors are able to produce a wide variety of products such as starch, dextrins, and high fructose syrup from the starch fraction that can also be used for ethanol production. These companies include: 1) ADM, 2) Cargill, 3) Staley, 4) Williams Bio-Energy, 5) Grain Processing Corp, and 6) CPC.

**1.1 Saccharification**

The dextrins (short chains of glucose 'monomers' resulting from the liquefaction process) need to be converted to glucose before the yeast can catalyze the glucose to ethanol reaction. Current state-of-the-art for dry mill ethanol generally incorporates saccharification during the fermentation (Simultaneous Saccharification & Fermentation). However, in the HS/LE process, the fermentation proceeds to completion in a very short time period, four to eight hours. Thus the conversion of the dextrins must be either finished prior to fermentation or completed within this same fermentation time period. In this project Bio-Process Innovation, Inc. will evaluate 1) 60 to 80% saccharification in a holding tank at 60° C prior to fermentation, and 2) use of an immobilized enzyme column where the dextrins are passed through the column and converted prior to being introduced to the HS/LE reactor.

*1.1.1 60-120 minute saccharification:* Glucoamylase will be added at two different levels (100% and 150% of enzyme company recommended levels) to batches of dextrins made up of 22-24% dextrins. The dextrins will be held at 55 to 60° C for various periods ranging from 30 to 200 minutes. The degree of saccharification will be measured and the syrup taken to the HS/LE reactor. Completion or non-completion of the conversion of the dextrins during eight-hour consecutive batch fermentation will be determined with various saccharification treatments. These results will be integrated with some standard modeling of the enzyme kinetics to determine optimal saccharification procedures.

*1.1.2 Immobilized enzyme column.* The ability of immobilized glucoamylase to convert dextrins has been demonstrated by a number of workers. Bio-Process Innovation, Inc. will test the procedures suggested by Lantero et al (1995), then test some improvements in carrier media. Work by Krishnan et al, (Biores. Tech. 75:99, 2000) suggests that a column of this nature should be regenerated after about three months. Based on the experimental results, modeling efforts will

be performed to determine the capital expenditures and possible savings associated with reducing glucoamylase usage.

*1.1.3 Process modeling and economics.* The two process alternatives for saccharification of dextrins will be compared to suggest the best system for new and retrofit corn ethanol plants.

### 1.2 Stillage Recycle

The ability of the BPI HS/LE system to handle a high recycle rate has been established on molasses, but not on corn-based glucose fermentations. In this portion of the project, BPI will run lab scale tests at 4 levels of 'stillage' recycle, 25%, 50%, 70% and 80% using a one L Multi-gen fermenter in the Consecutive Batch Mode. The experiment will run approximately three batches/day (eight hour fermentations) for ease of scheduling. These trials will be integrated with the saccharification trials of Task 1.1.1. (free enzyme). Dried dextrins will be made up with the desired degree of back-set prior to saccharification. The back-set is prepared by stripping the ethanol from a previous batch of 'beer' using a vacuum stripping procedure to simulate the distillation process. Ethanol levels will be reduced to less than five g/L (0.5%) Each level of stillage recycle will be tested over a period of 10 to 20 consecutive batch cycles to demonstrate the stability of the process at the various recycle percentages. Brix will be measured to determine the reaction rate and samples will be taken when the reaction is near completion. Samples will be analyzed for glucose, ethanol, dextrins, and level of glycerol and lactic acid. Glycerol is the major by-product of the ethanol fermentation, and the glycerol levels will increase as the fraction of backset increases. These trials will establish the inhibitory effects of different levels of backset, and an 'optimal' level will then be selected for a pilot scale demonstration. This optimal level is a level that maximizes back-set while not causing a large degree of inhibition in the reactor so that the reaction still completes in less than eight hours in the Consecutive Batch Mode.

### 1.3 HS/LE Pilot

A 300 to 5000-gallon fermenter will be designed, fabricated, or modified for the HS/LE fermentation. This bio-reactor will then be used first for the Wet Mill Syrup trials, then during the last two quarters of the project operated on dry mill syrup.

*1.3.1. Siting agreement.* Several possible sites for installation and testing of the HS/LE bioreactor have been identified. These include the National Corn-to Ethanol Pilot Plant in Illinois, a dry mill ethanol producer in Minnesota, a small ethanol producer in Iowa, and a Broin plant in Michigan. These discussions will be completed and a siting agreement written. The discussions will identify the size of the HS/LE reactor.

*1.3.2. Detailed design of the HS/LE bioreactor.* This reactor will be:

- i. fitted with a variable speed stirring propeller,
- ii. fitted with an external shell and tube heat exchanger for controlling the temperature,
- iii. fitted with small 'sight glasses' and sample ports to evaluate the size and nature of the flocs over time,

The use of a low-speed, centrifugal pump for circulation of the fermentation broth through the HX will be tested to see if the shear forces in the pump negatively affect the yeast pellets. The use of a positive displacement pump may be required if the centrifugal pump is too disruptive to the pellets.

The pilot plant will be designed to allow sterilization of the reactor with low-pressure steam (two psig) and introduction of a start-up culture from a 200 L culture vessel.

Complete construction diagrams of the reactor, controls, and P&ID diagram for the pilot will be completed.

*1.3.3 Fabrication of the HS/LE bioreactor.* The reactor will be built during Q3 of the project.

*1.3.4 Installation of the Bioreactor.* The HS/LE reactor will be installed at the site host during Q3 of the project. Tanks for make-up of the feedstock will be sized to allow 6 to 10 batches.

*1.3.5 Operation of the HS/LE Bioreactor on wet mill corn syrup.* BPI will operate the pilot plant for a period of 30 days in the Consecutive Batch Mode. For the first trials, no stillage recycle will be used in preparing the feed while, if practicable on the pilot scale, stillage recycle at the optimal level determined from lab scale studies (4.1.b) will be run for the final week to 10 days of operations.

## **Task 2. Dry Mill HS/LE**

### **2.1 Effect of Insoluble Solids**

During Q3 and Q4, BPI will be performing lab and pilot-scale tests with centrifuges to determine the required processing equipment to give the required clarity in the dry mill dextrins fed to the HS/LE fermenter from a solids washing centrifuge as per BPI's DM-2 process. In this portion of the project, the project will determine at what concentration Non-Soluble Solids (NSS) interfere with the long term performance of the HS/LE process. Dry-milled 'cooked' mash will be screened and rinsed using a 500 micron screen to separate the soluble dextrins from the corn fibers/NSS. The level of NSS in the liquid fraction will be measured and correlated with the optical density (OD) of the dextrin syrup. TEMA will further provide BPI samples of the thin stillage from their Conturbex rinsing centrifuges currently used in the corn dry milling ethanol plants (if permission for the samples is given by the user) to get an idea of the sort of NSS provided by a rinsing centrifuge. The effect of various sized bowl screens on the NSS carry-through in to the syrup will be evaluated. The syrup will be further clarified if required based on data gathered from lab experiments. Some trials will be run using a small scale centrifuges (i.e. H-320) The syrup will be centrifugally clarified to determine the ability of a 'desludging' (high speed disk/cone) centrifuge to separate the residual NSS. Trials will be run on the 1 L Multigen with NSS from the dry mill screenings at various levels of NSS.

### **2.2. Determination of Separation Technologies**

Determination of separation technologies for required level of suspended solids were determined in 2.1. Based on the results of 2.1, different separation strategies will be evaluated if required to bring the NSS levels in the dextrin syrup down to levels acceptable by the HS/LE fermentation. These may include 1) desludging centrifuge: this type of centrifuges is also termed a 'clarifier' in the dairy industry and has the ability to give a separation of 5 to 10,000 G force over a short (disk plate) separation space and 2) membrane separation: there are cross flow membrane systems which would give an 'absolute' separation based on particle size. Membranes with 0.2 to 0.45 micron are often used for 'sterilization'.

### **2.3 HS/LE pilot**

*2.3.1 Pilot Plant Design/Specifications* Based on results of 2.1 and 2.2, a detailed P&ID of the DM-2 pilot scale process will be completed.

*2.3.2 Installation of Pilot Eq.* The equipment specified in Task 2.3.1 will be assembled and installed at the host site during Q5 and Q6.

*2.3.3 Operation of Pilot HS/LE with Dry mill syrup.* The 1000 to 2000 gallon pilot plant fermenter used for the wet mill demonstration of the HS/LE will be used for the dry mill demonstration in Q7

and Q8. Dry mill mash (prepared in a batch cook process) will be separated into 'clear' dextrin syrup and a corn solids stream using a pilot scale worm-screen type rinsing centrifuge provided/leased to the project by TEMA.

### **Task 3. MVR Distillation**

As the final portion of this project, BPI will complete thorough design, optimization, and costing associated with implementing the MVR technology, both as a retro-fit and for new construction at 15, 30, and 60 million gallon/year scales.

#### **3.1 Modeling/Optimization**

A stage-by-stage model of the ethanol distillation using the Van Laar model for ethanol-water activity coefficients as a function of temperature and composition (Dale et al, 1985) will be used to model the performance of the column. Boiling points as a function of stillage recycle will be included as a process parameter. BPI has an extensive background with ethanol distillation and dehydration. This background will be combined with engineering specifications on structured packing data provided by Norton, Koch, and Lantec companies – makers of column packing. Optimized variables include type of packing, operating pressure of column, and approach temperature across the reboiler (a closer approach temperature requiring more Heat Exchanger (HX) area, but allowing a lower vapor recompression ratio and less compressor HP).

#### **3.2 Detailed Design/Controls**

A detailed engineering diagram of a BPI MVR distillation column with a capacity of 20 million gallons/year based on a 12% (V/V) ethanol beer feed will be completed. Costs and performance of the compressor will be compared to data provided by compressor manufacturers (MD, Atlas Copco, Alfa Laval). These prints and P&ID diagram of the process will be completed during Q3 and Q4 of the project and hopefully lead to actual installation of a unit in the later phases of the project.

### **Task 4. Project Management and Reporting**

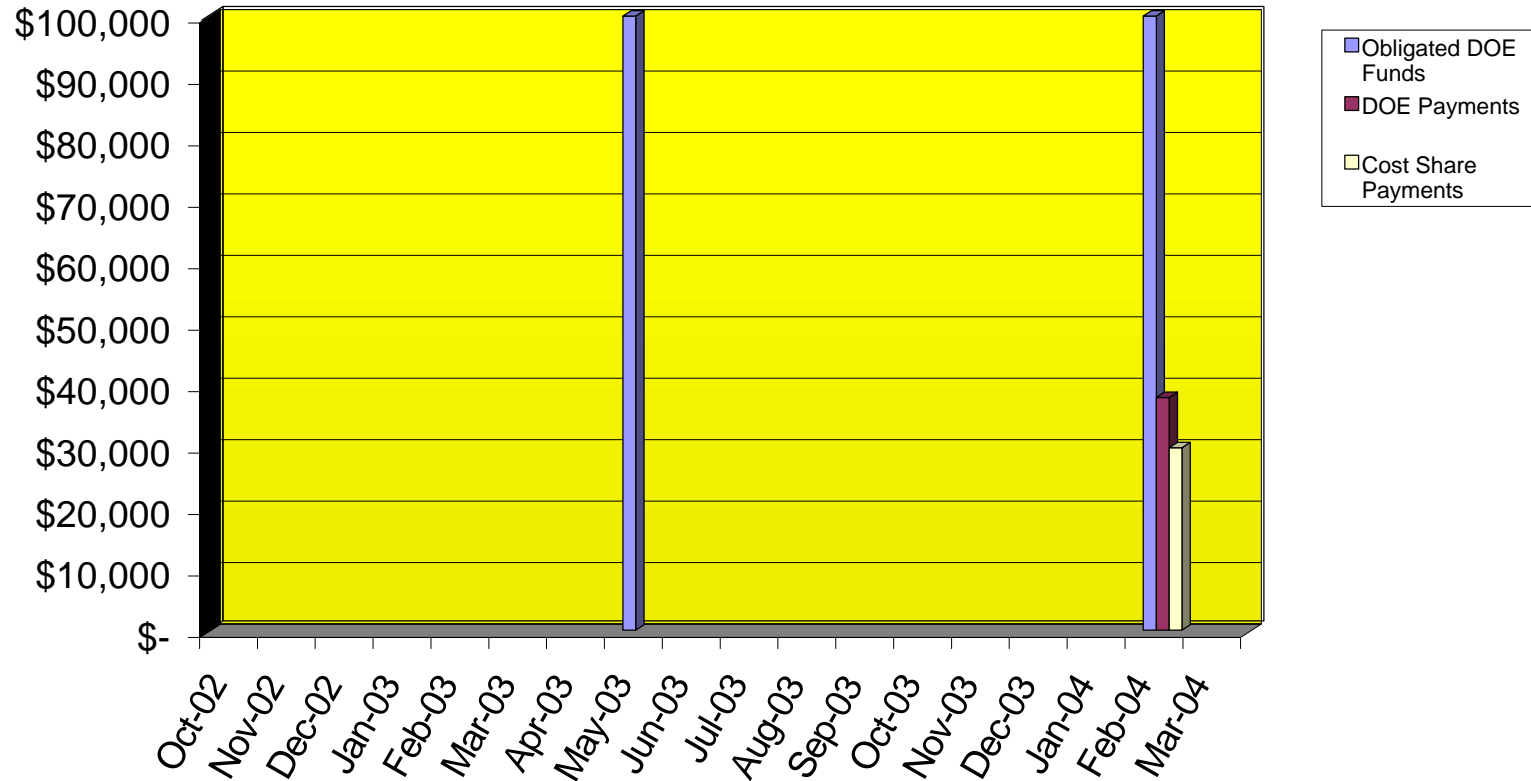
BPI is responsible for submitting both Semi-Annual Progress Reports and a Final Report to DOE. The Semi-Annual Reports are due every April 30 and October 31. The Final Report is due 90 days after the project completion date as specified in the agreement. This task also includes other DOE requirements for market assessments, fact sheets, benefits analyses, workshops, etc.

## Project Cost Performance in DOE Dollars for Fiscal Year 2003

DE-FG36-03GO13006

Bio-Process Innovation, Inc.

High Speed / Low Effluent Process for Wet and Dry Mill Corn to Ethanol and Co-Products



	Oct-02	Nov-02	Dec-02	Jan-03	Feb-03	Mar-03	Apr-03	May-03	Jun-03	Jul-03	Aug-03	Sep-03
Obligated DOE Funds	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$100,000	\$0	\$0	\$0	\$0
DOE Payment	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Cost Share Payment	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0

	Oct-03	Nov-03	Dec-03	Jan-04	Feb-04	Mar-04	PFY*	Cumulative
Obligated DOE Funds	\$0	\$0	\$0	\$0	\$100,000	\$0	\$0	\$200,000
DOE Payment	\$0	\$0	\$0	\$0	\$37,899	\$0	\$0	\$37,899
Cost Share Payment	\$0	\$0	\$0	\$0	\$29,708	\$0	\$0	\$29,708

Approved DOE Budget:	\$200,000
Approved Cost Share Budget:	\$410,634
Total Project Budget:	\$610,634

\* Prior Fiscal Years



Bio-Process Innovation, Inc. - 03GO13006																																						
ID	Task Name		2004												2005												2006											
			Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May			
1	Task 1.0: HS/LE with Wet Mill Syrup		<div><div></div></div> 100%																																			
2	Task 1.1: Saccharification		<div><div></div></div> 90%																																			
3	Task 1.2: Stillage Recycle		<div><div></div></div> 90%																																			
4	Task 1.3: Pilot Plant		<div><div></div></div> 5%																																			
5	Task 2.1: Effect of Insoluable Solids														<div><div></div></div> 5%																							
6	Task 2.2: Solids Seperation Technology														<div><div></div></div> 0%																							
7	Task 2.3: Pilot Plant														<div><div></div></div> 0%																							
8	Task 3.1: Modeling/Optimization		<div><div></div></div> 70%																																			
9	Task 3.2: Detailed Engineering of Column		<div><div></div></div> 25%																																			
10	Task 4: Project Management		<div><div></div></div> 45%																																			